In Situ Neutron and Synchrotron X-ray Diffraction Studies of NiTi-based High Temperature Shape Memory Alloys

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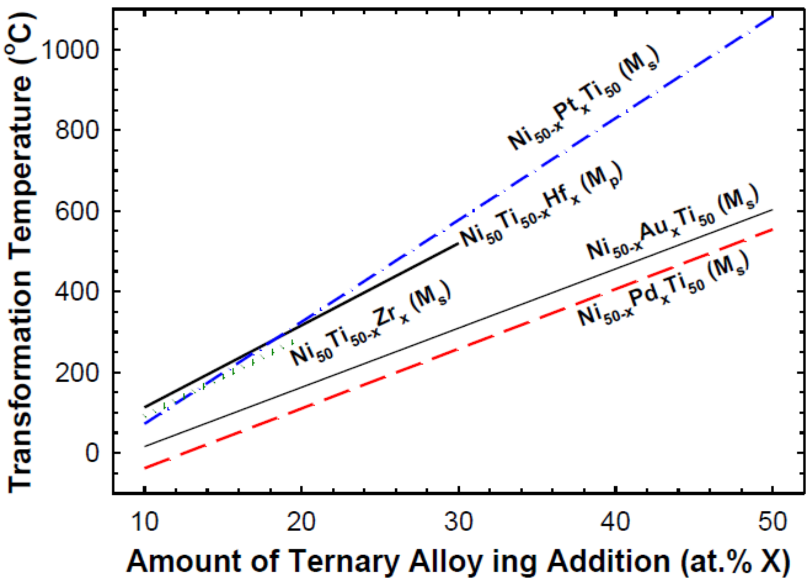
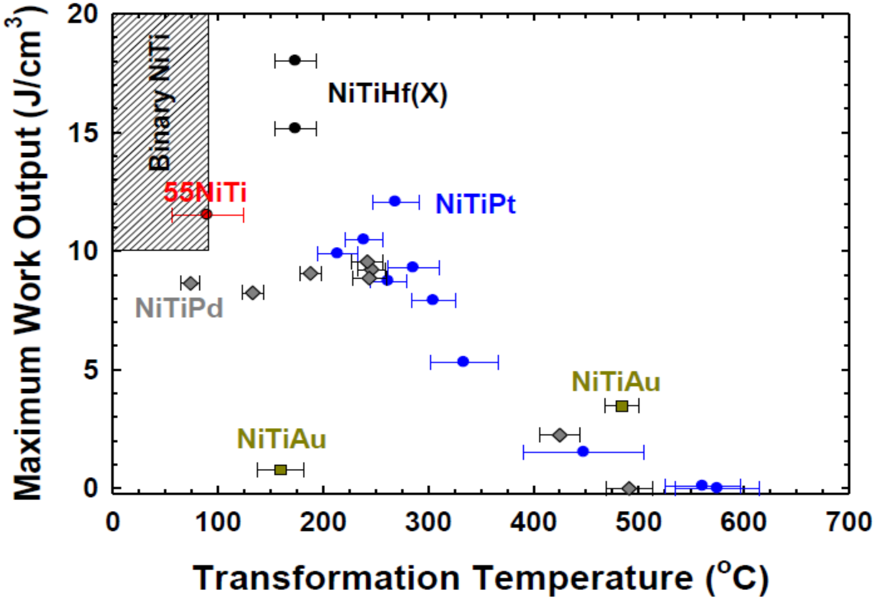
High Temperature Shape Memory Alloys (HTSMAs)

- ➢ **Part of SMA research at NASA GRC is directed toward the development of HTSMAs, understanding and predicting their macroscopic and microstructural behavior, and introducing them into large scale commercial devices.**

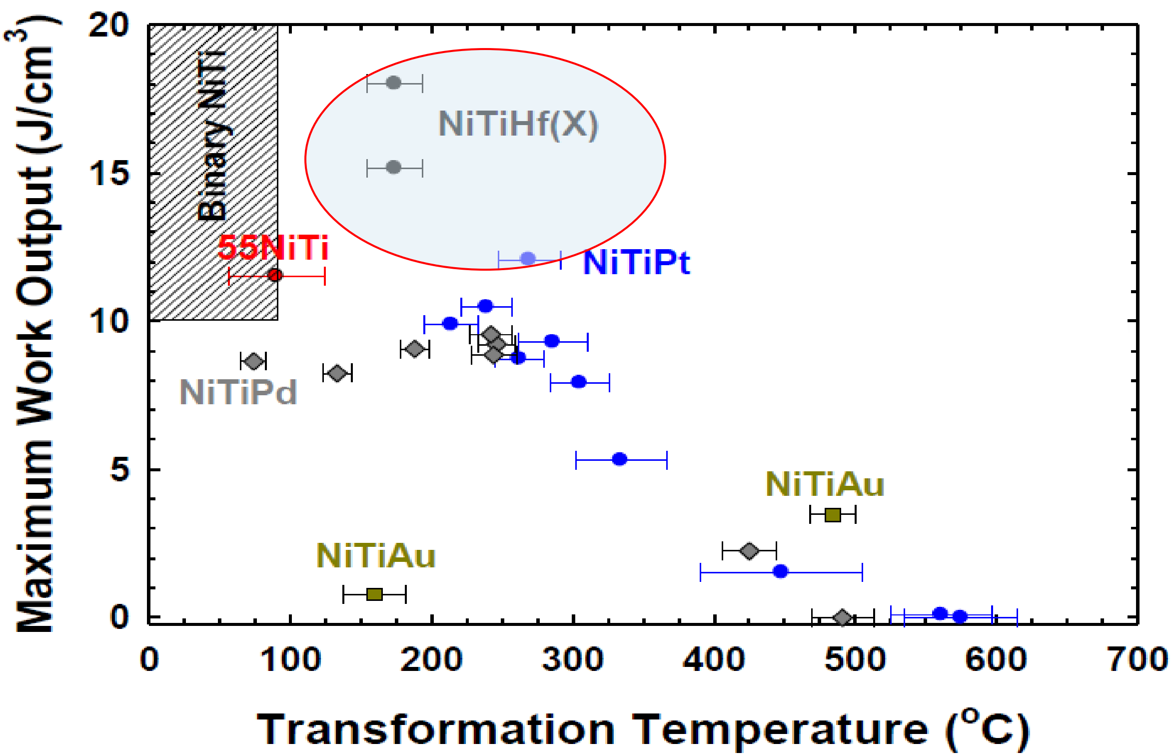




<u>Objectives:</u>

- ➢ **Targeted HTSMA development to meet device requirement**
- ➢ **To do that, we must provide links between the macroscopic behavior and the underlying micromechanics (*in situ* neutron and synchrotron X-ray Diffraction)**
- ➢ **Extension to low temperature and cryogenic SMAs**

Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$)

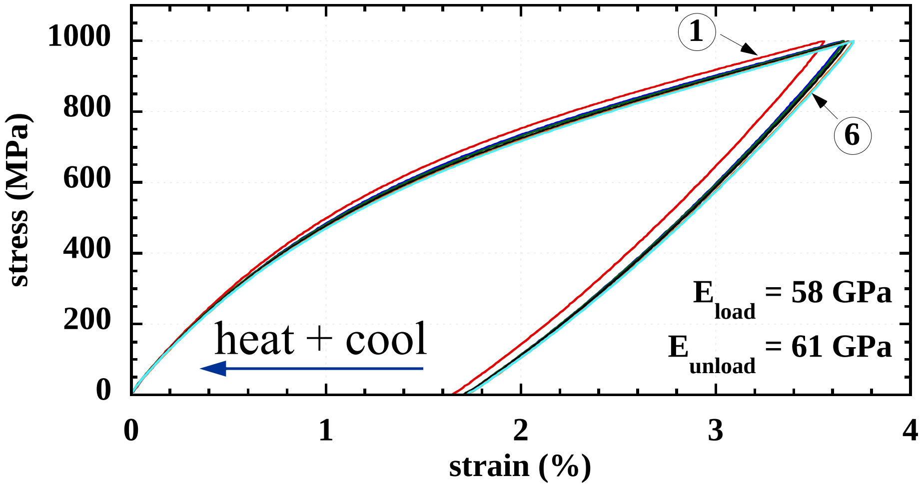


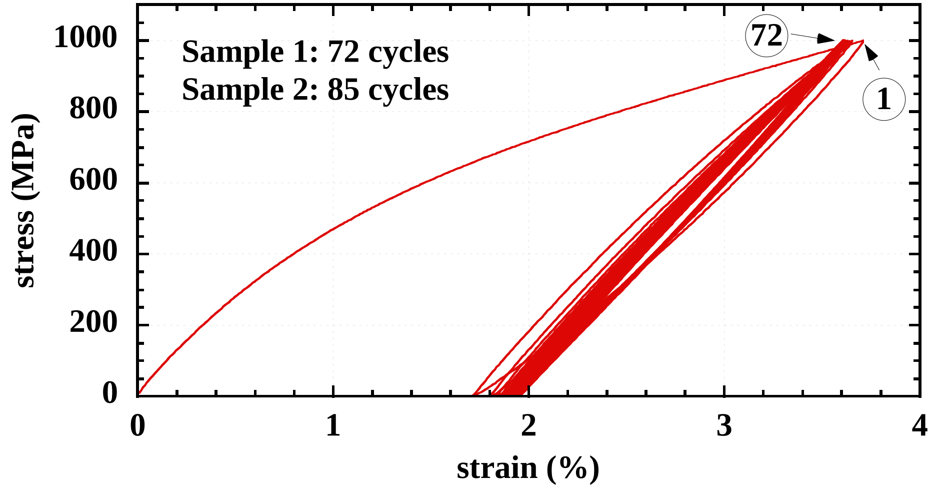
Extruded and aged NiTiHf

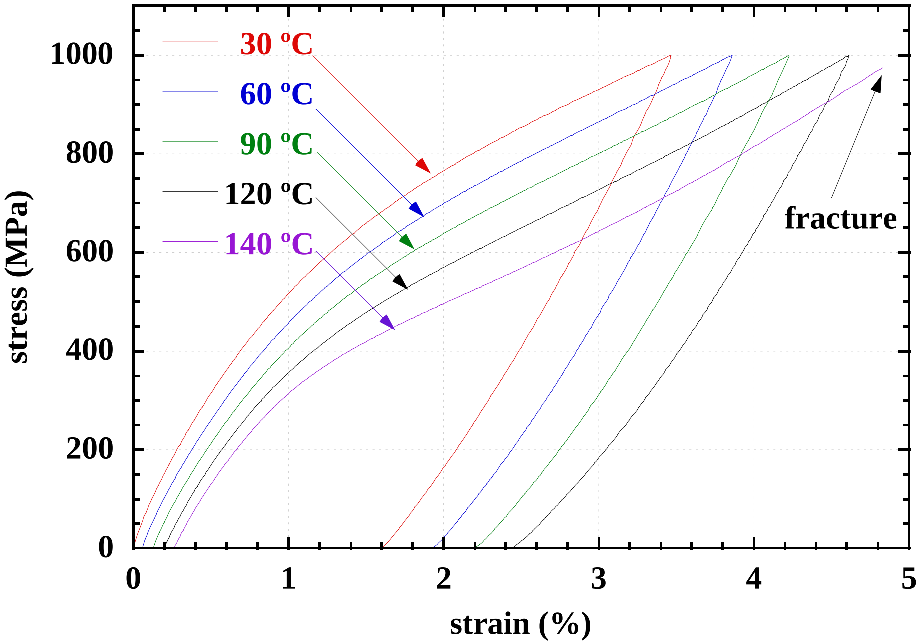
- **Why Hf?**
  - **HTSMA (No precious metals)**
  - **Af > 150 ºC (can be modified to lower temperatures)**
  - **Little or no training required (inherent dimensional stability)**

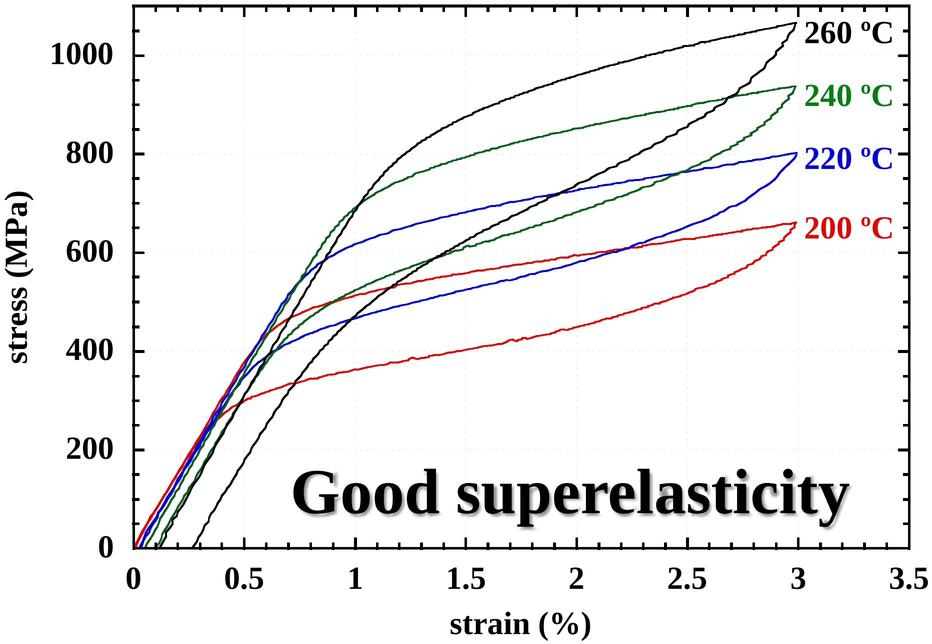
Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isothermal Response

No plastic strain up to the tested 1GPa



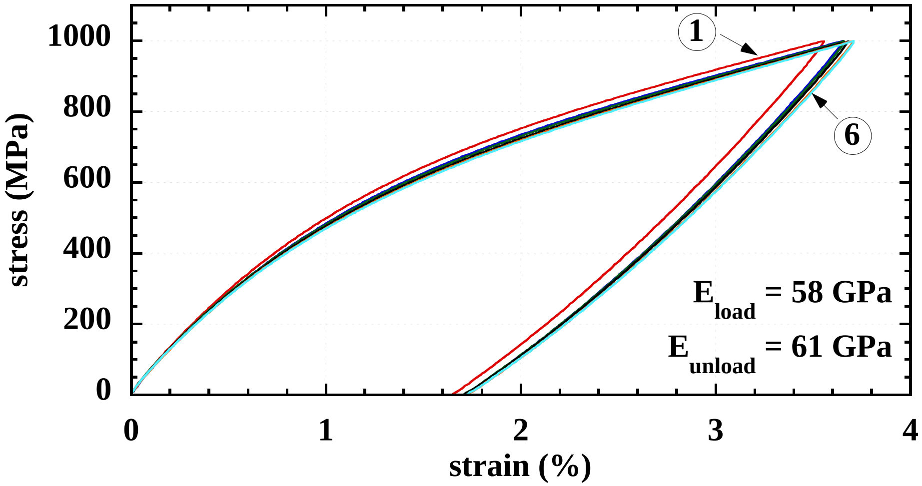


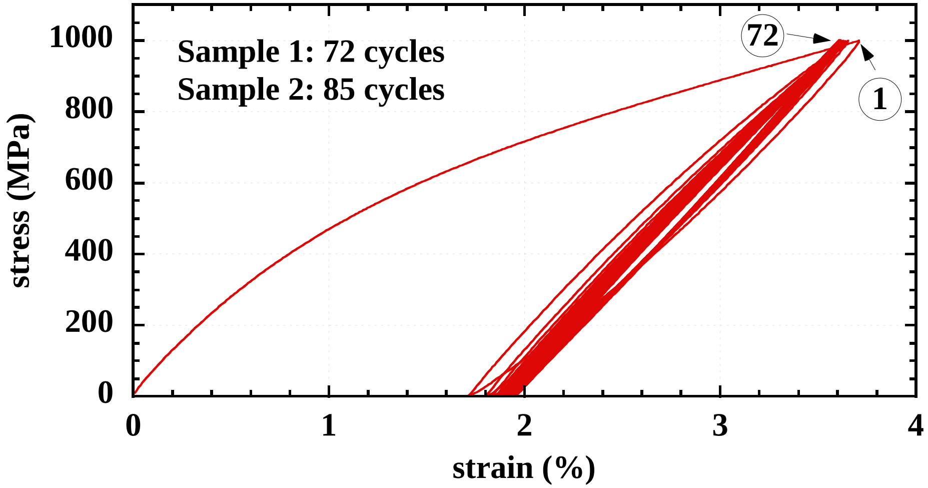


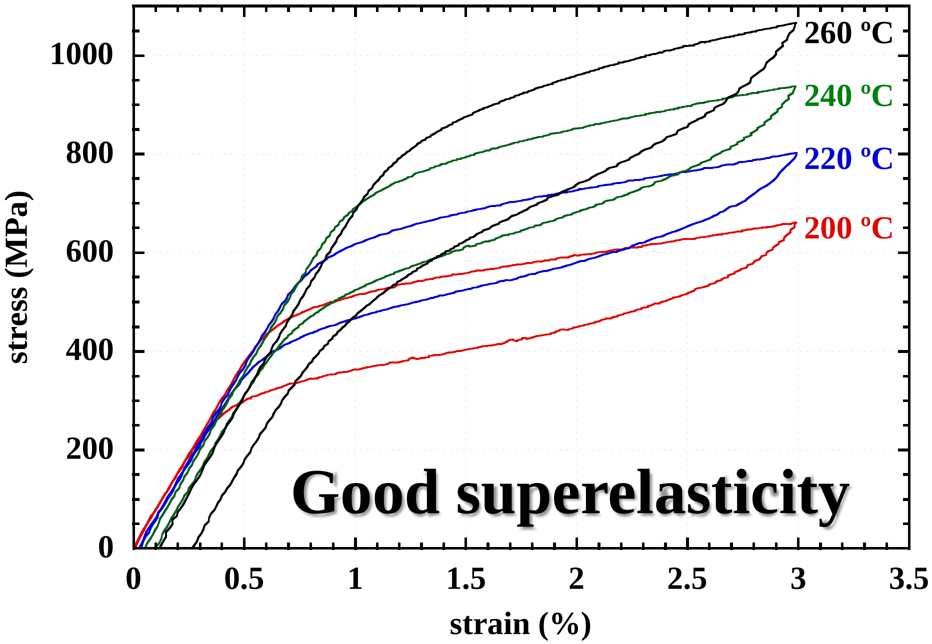


Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isothermal Response

No plastic strain up to the tested 1GPa

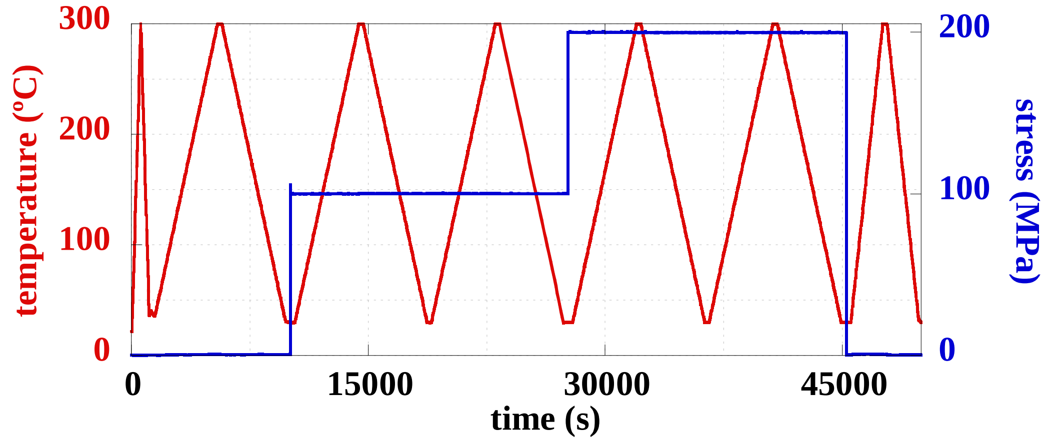


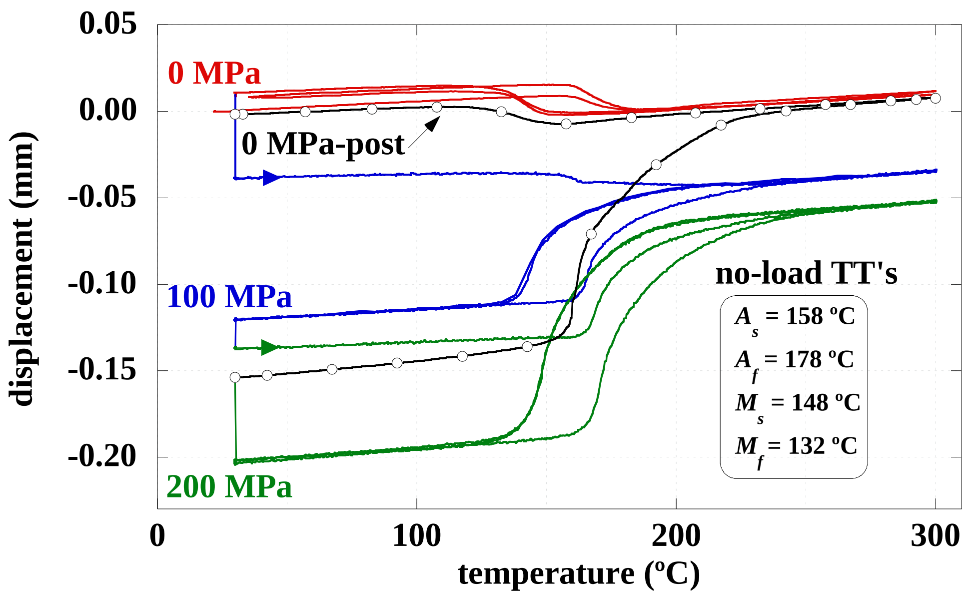




Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (C)

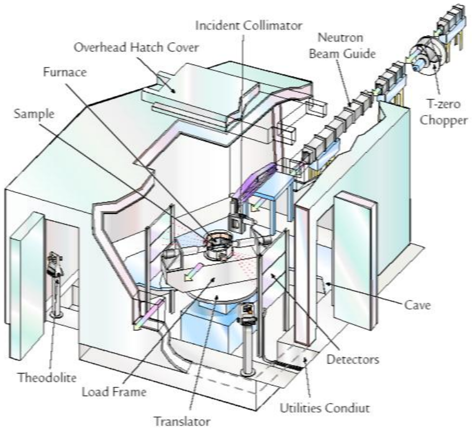
Macroscopic

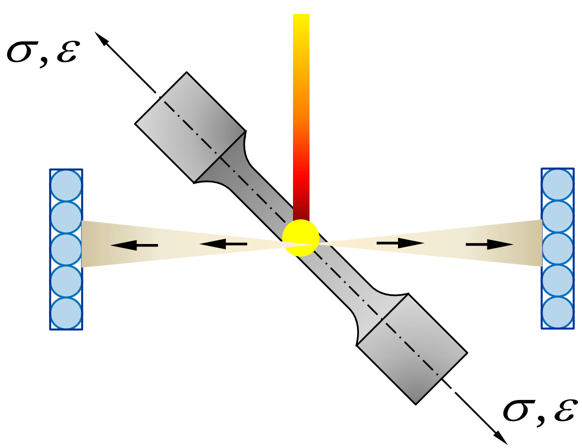




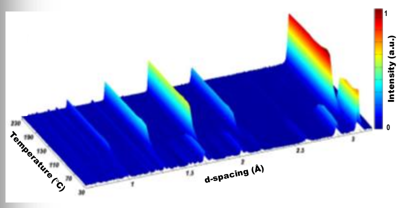
In situ Diffraction

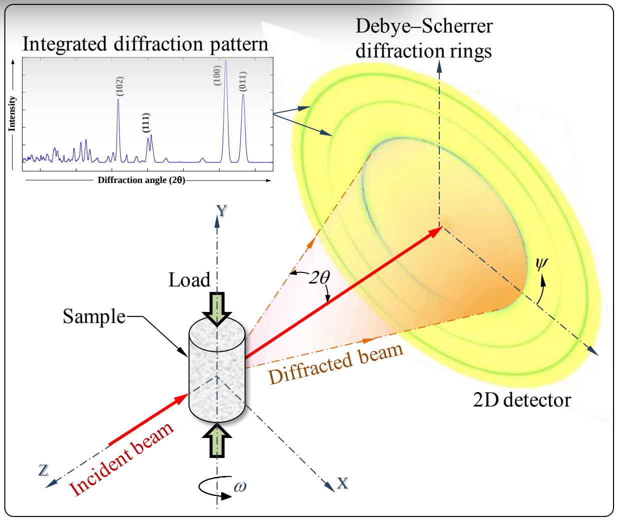
NEUTRON DIFFRACTION
Los Alamos National Laboratory (LANL)
Spectrometer for MAterials Research at Temperature and Stress (SMARTS)





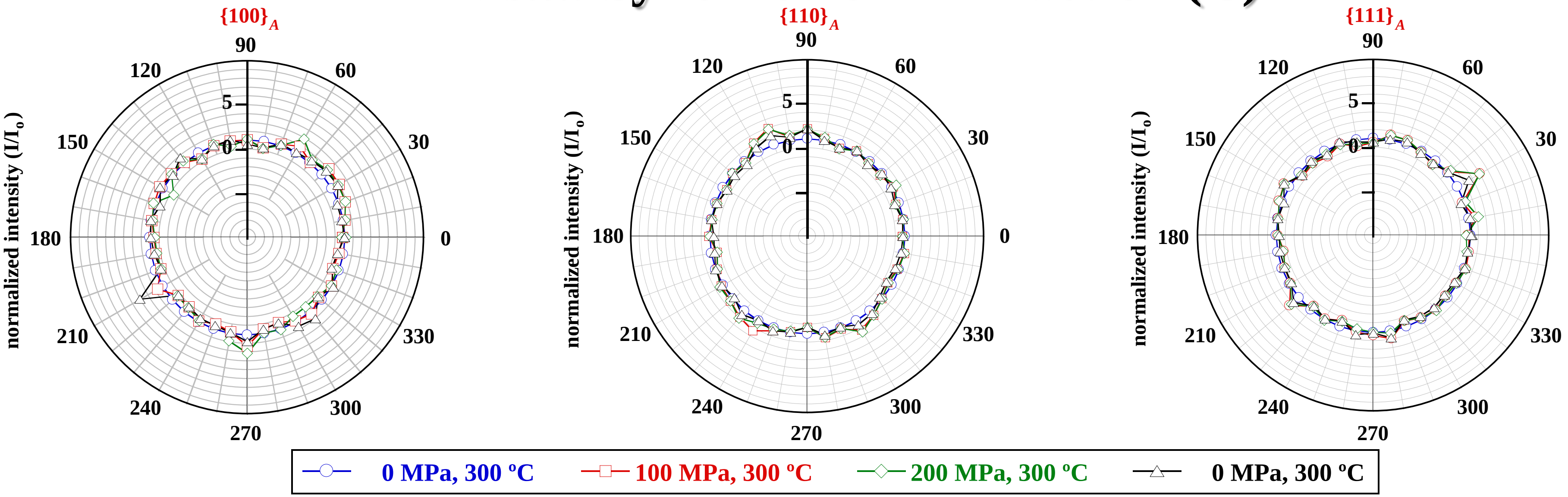
SYNCHROTRON X-RAY DIFFRACTION
Helmholtz-Zentrum Geesthacht (PETRA III)
High Energy Materials Science Beamline (HEMS)



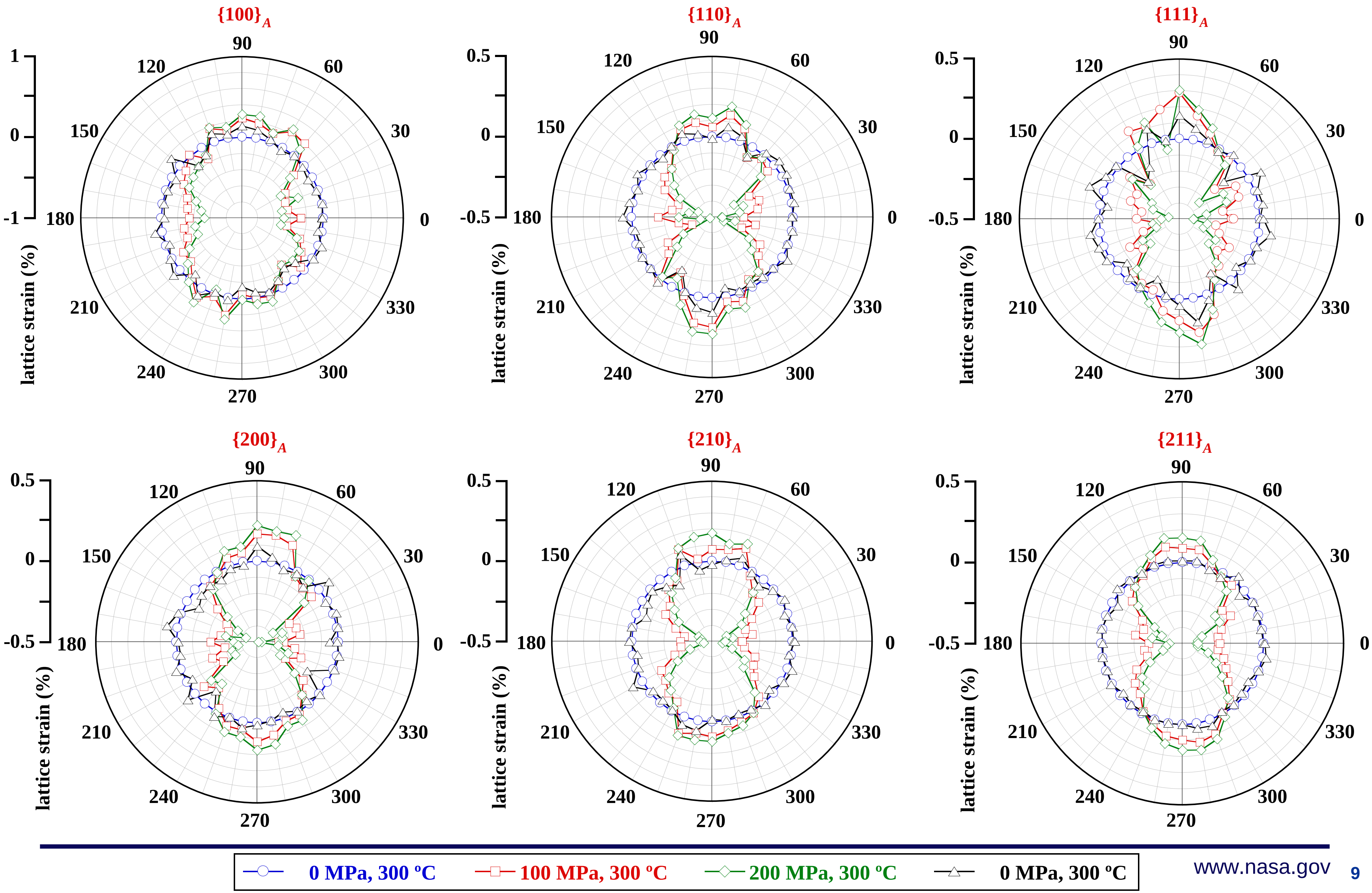


Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B2)

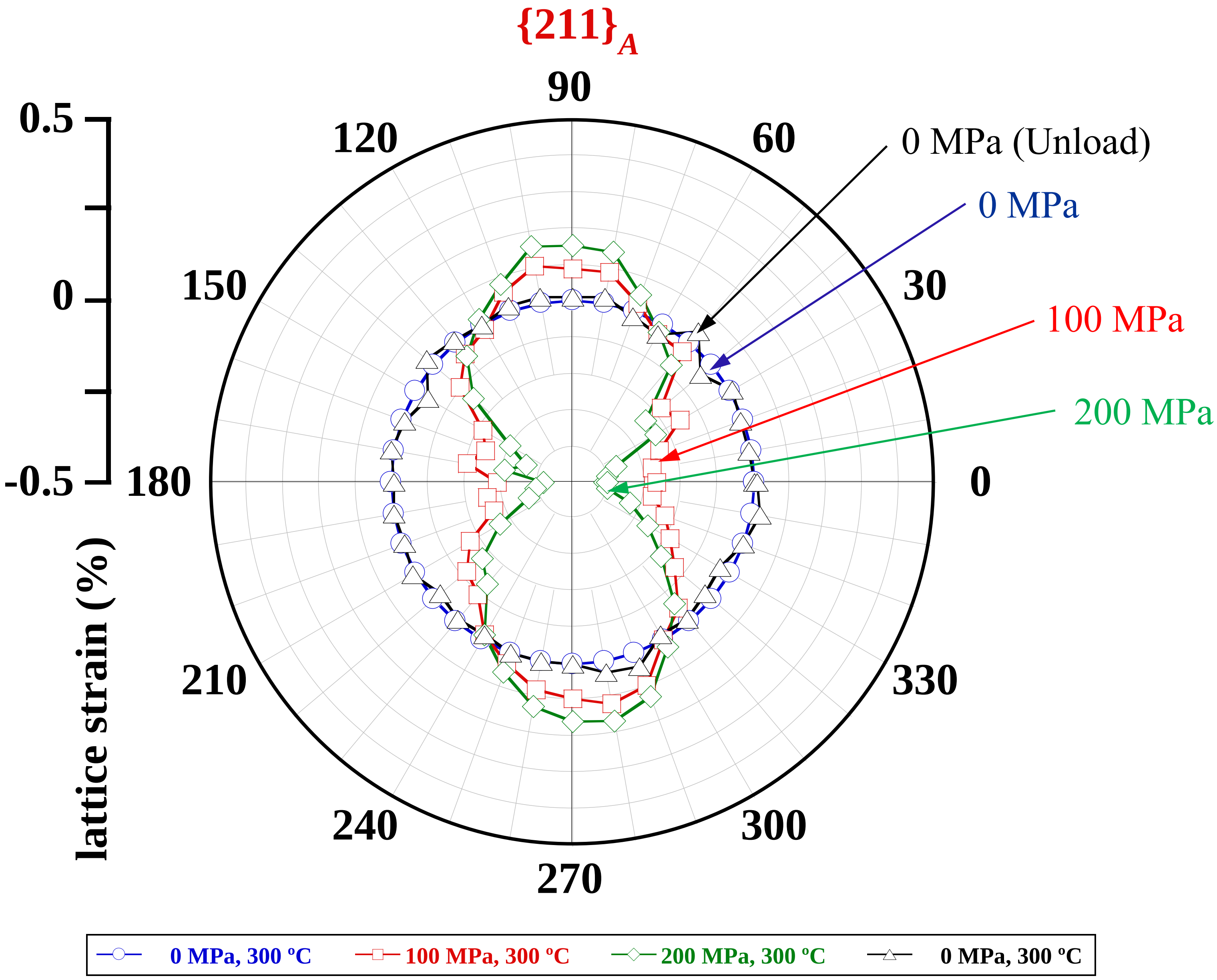
In situ Synchrotron Diffraction (C)



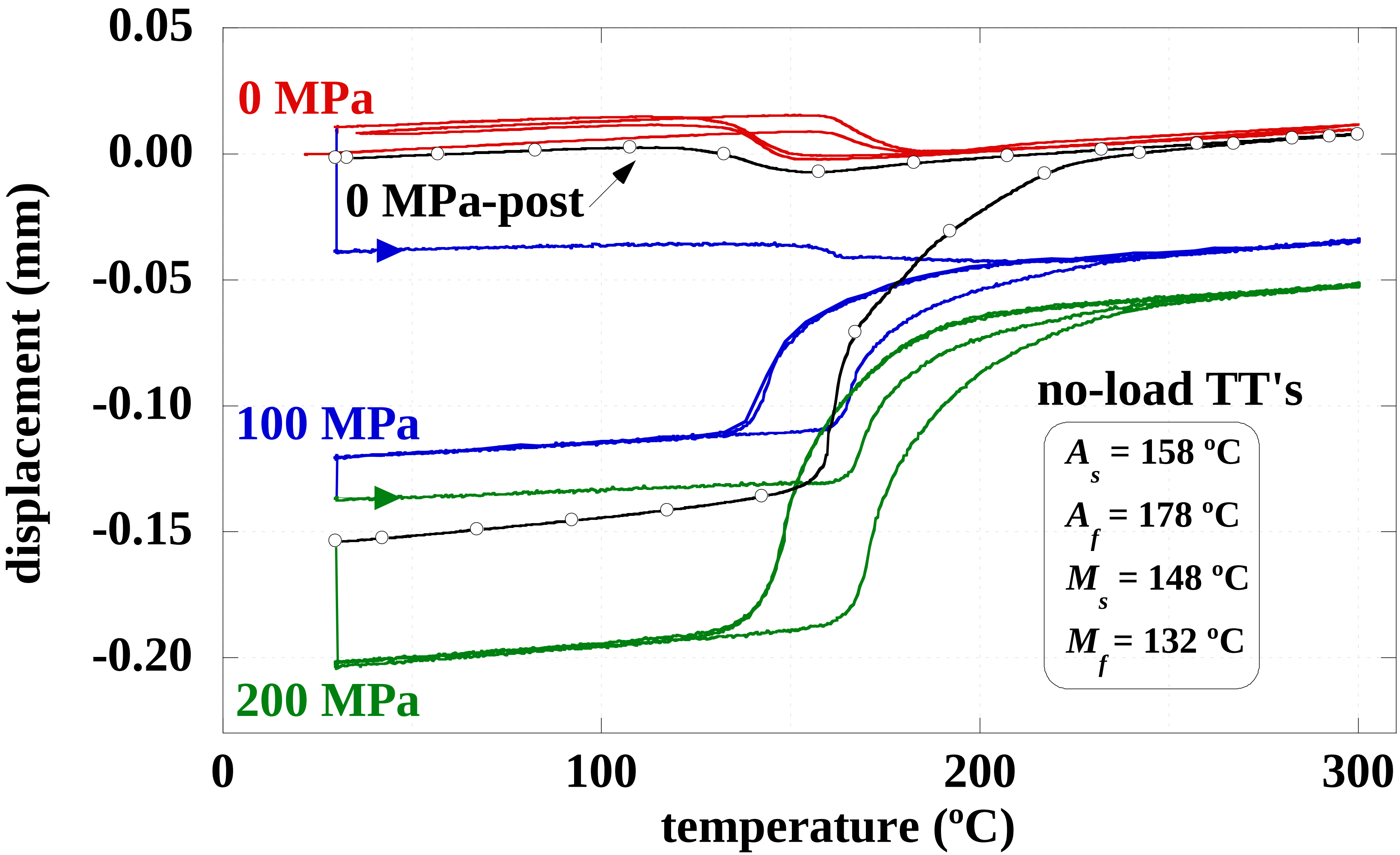
Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B2)
No Plastic Strain



Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B2)

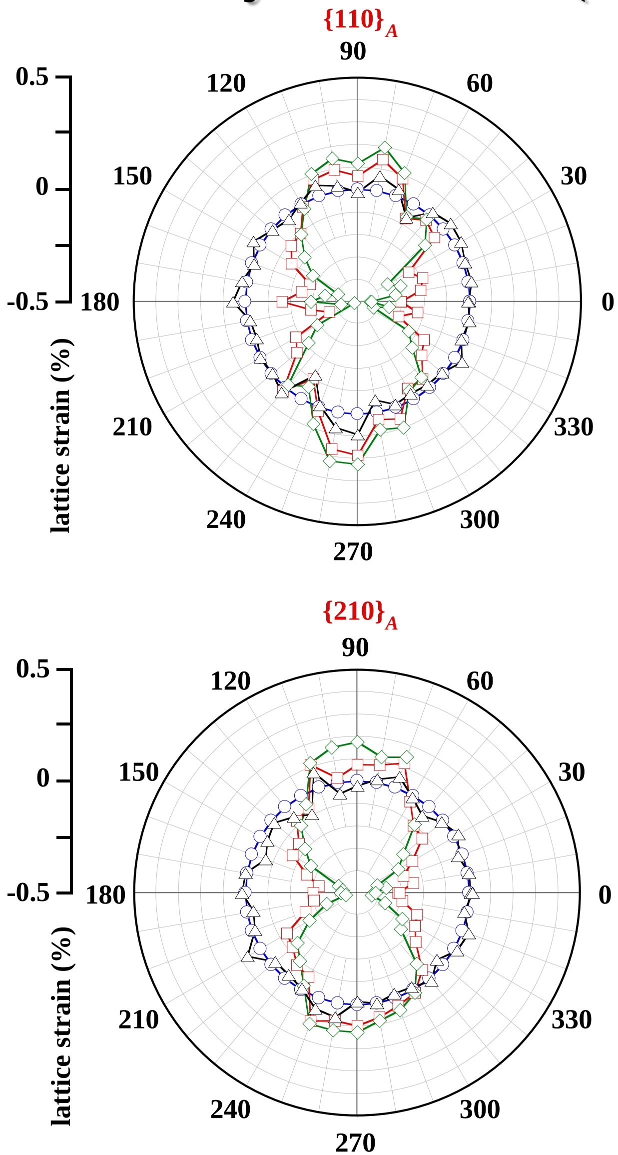


Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B2)

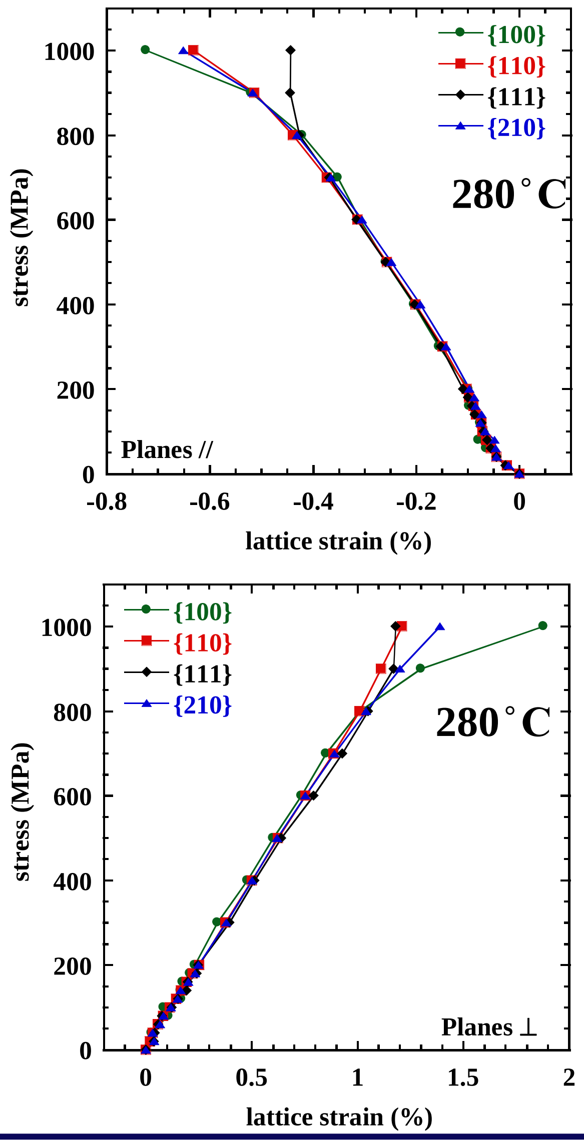


Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B2)

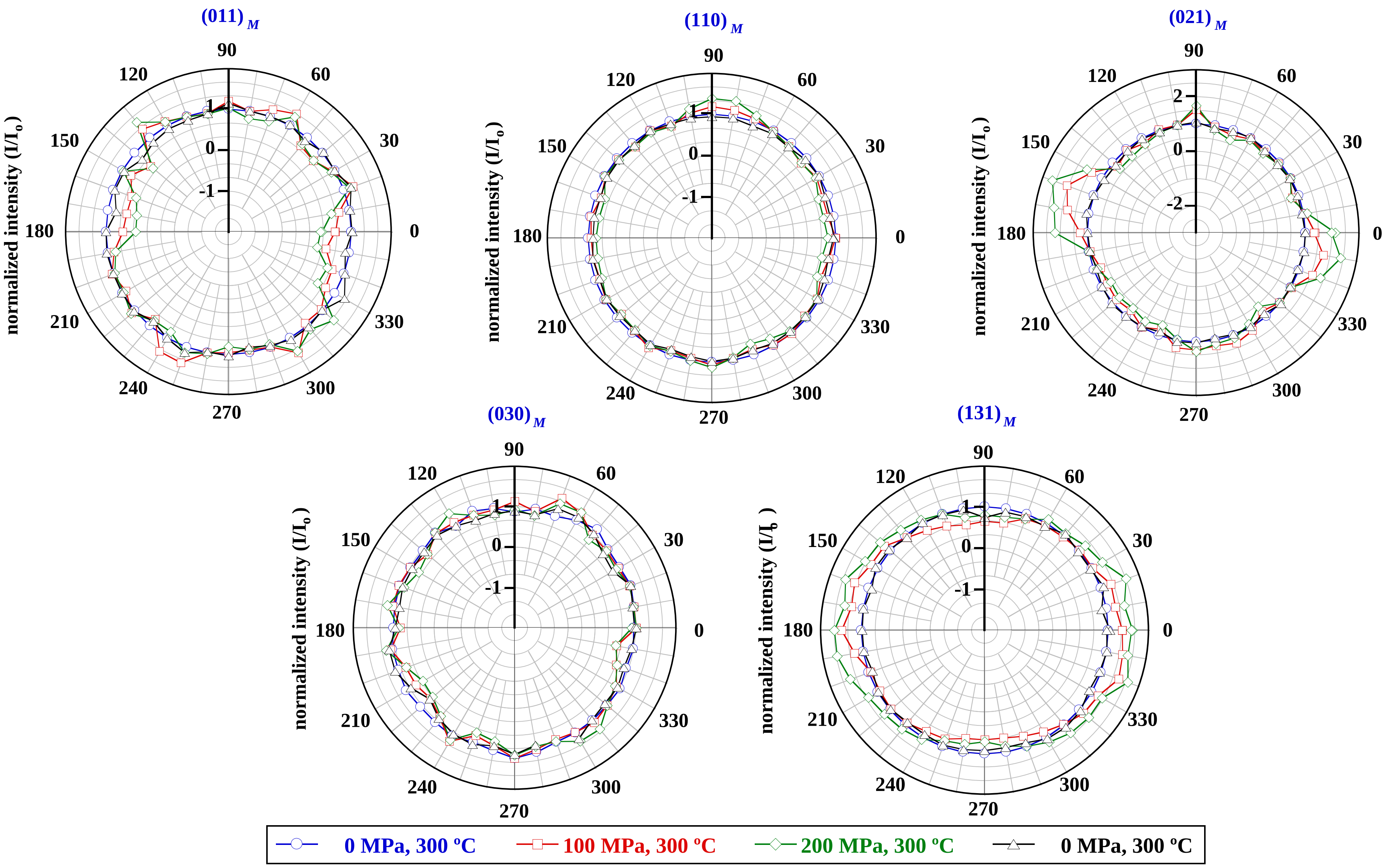
In situ Synchrotron (C)



In situ Neutron (T)



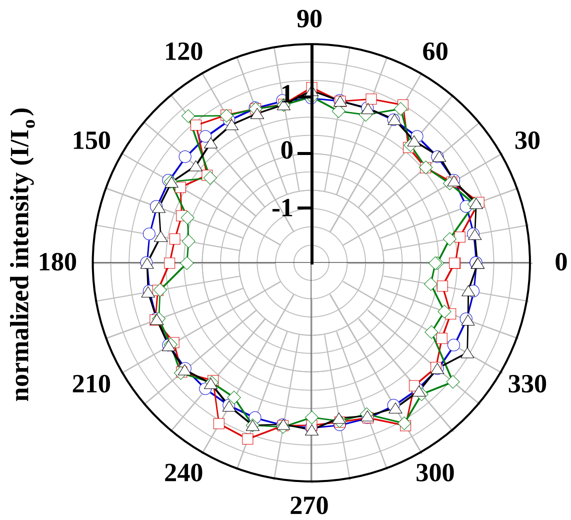
Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B19') Texture Evolution in Martensite



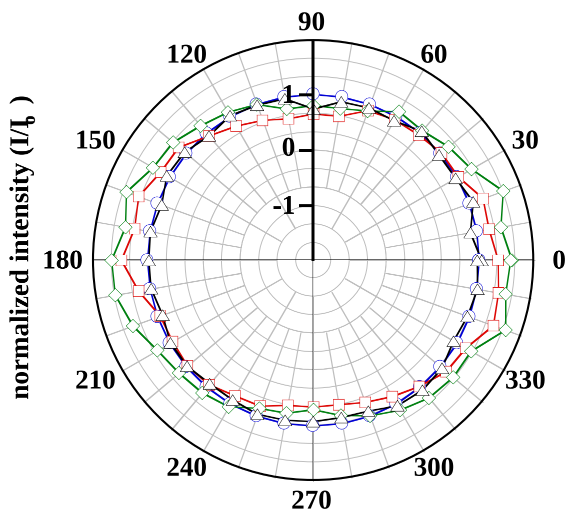
Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Isobaric Response (B19')

In situ Synchrotron (C)

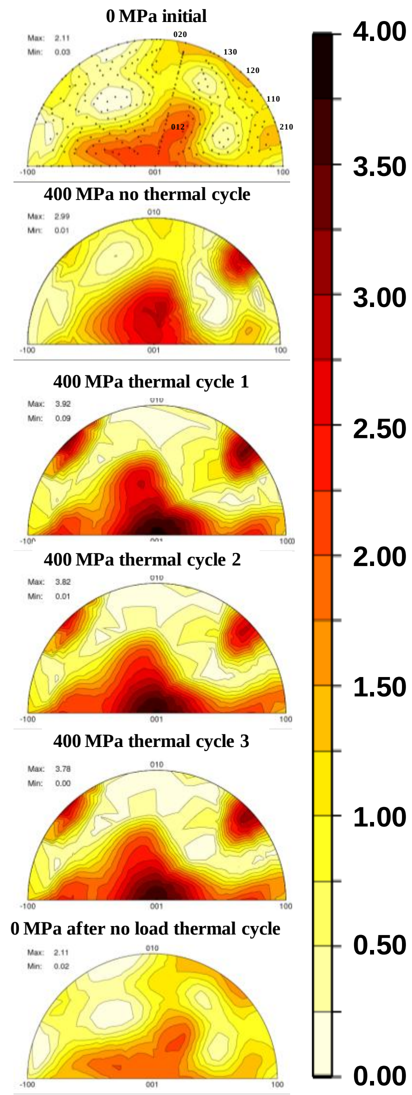
$(011)_M$



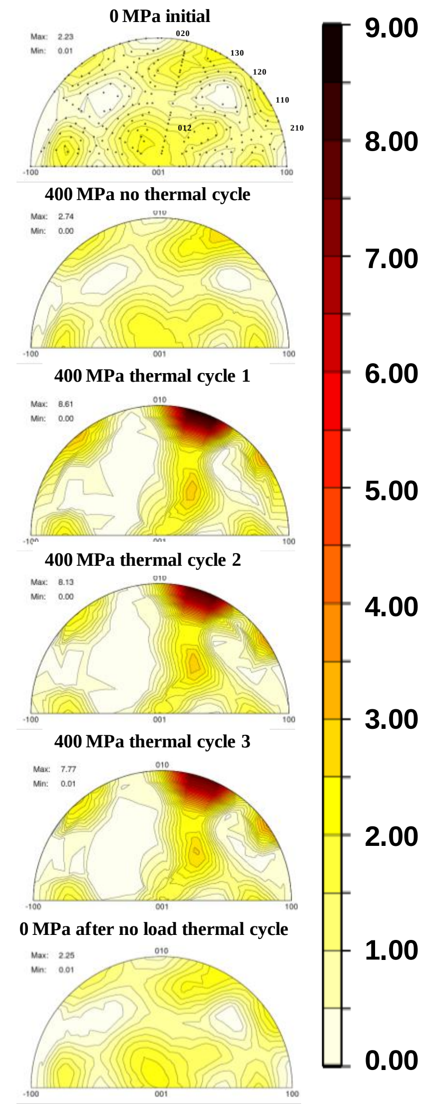
$(131)_M$



In situ Neutron (T)



(a)



(b)

Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$) Summary Precipitates are Key

SEM

- Fine, nanometer size, coherent precipitate phase (through stoichiometry control and aging)
- Limited detwinning attributed to the pinning of twin and variant boundaries by the dispersion of fine precipitates
- Efficient obstacles to irreversible plastic deformation
- Precipitate phase is believed to be the stabilizing factor in this alloy

Ni-Rich ($Ni_{50.3}Ti_{29.7}Hf_{20}$)- Literature

Microstructural Response During Isothermal and Isobaric Loading of a Precipitation-Strengthened Ni-29.7Ti-20Hf High-Temperature Shape Memory Alloy

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METALLURGICAL AND MATERIALS TRANSACTIONS A VOLUME 43A, DECEMBER 2012—4539

Fan Yang; Daniel R Coughlin; Patrick J Phillips; Limei Yang; Arun Devaraj; Libor Kovarik; Ronald D Noebe; Michael J Mills **Structure analysis of a precipitate phase in a Ni rich high temperature NiTiHf shape memory alloy,** *Acta Mat., accepted*

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Scripta Materialia 64 (2011) 725–728
Scripta MATERIALIA
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Load-biased shape-memory and superelastic properties of a precipitation strengthened high-temperature $Ni_{50.3}Ti_{29.7}Hf_{20}$ alloy

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Characterization of the microstructure and mechanical properties of a 50.3Ni–29.7Ti–20Hf shape memory alloy

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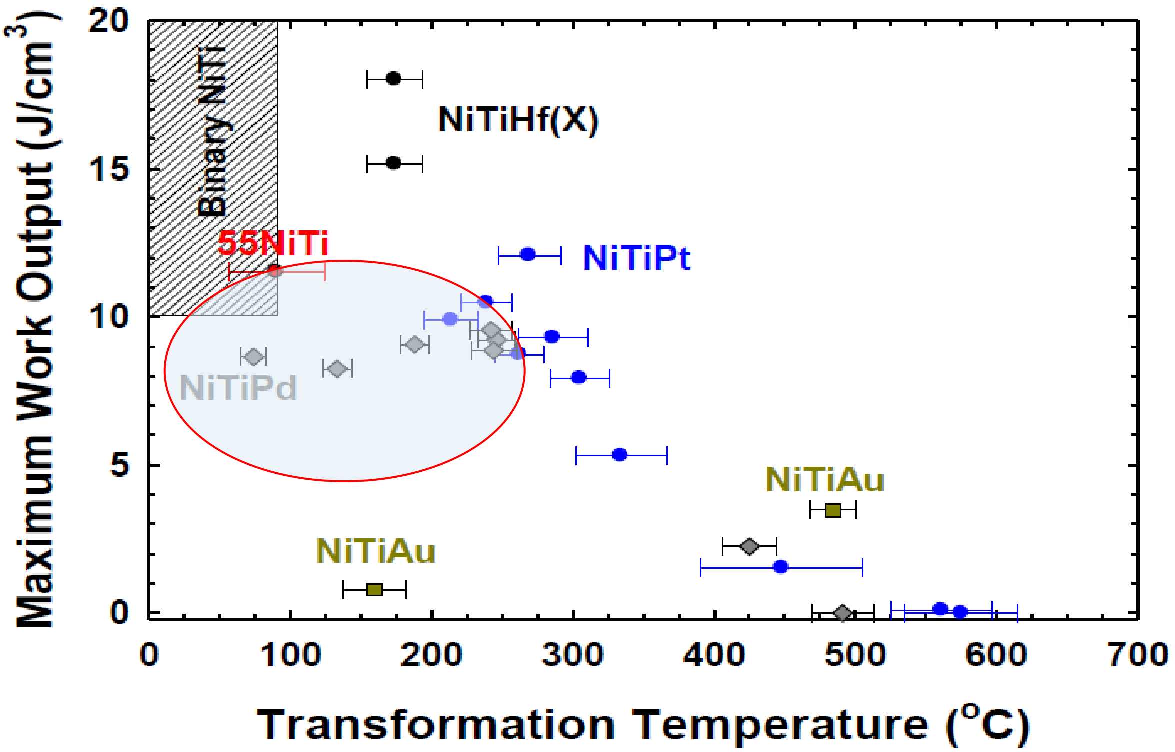
Available online 3 April

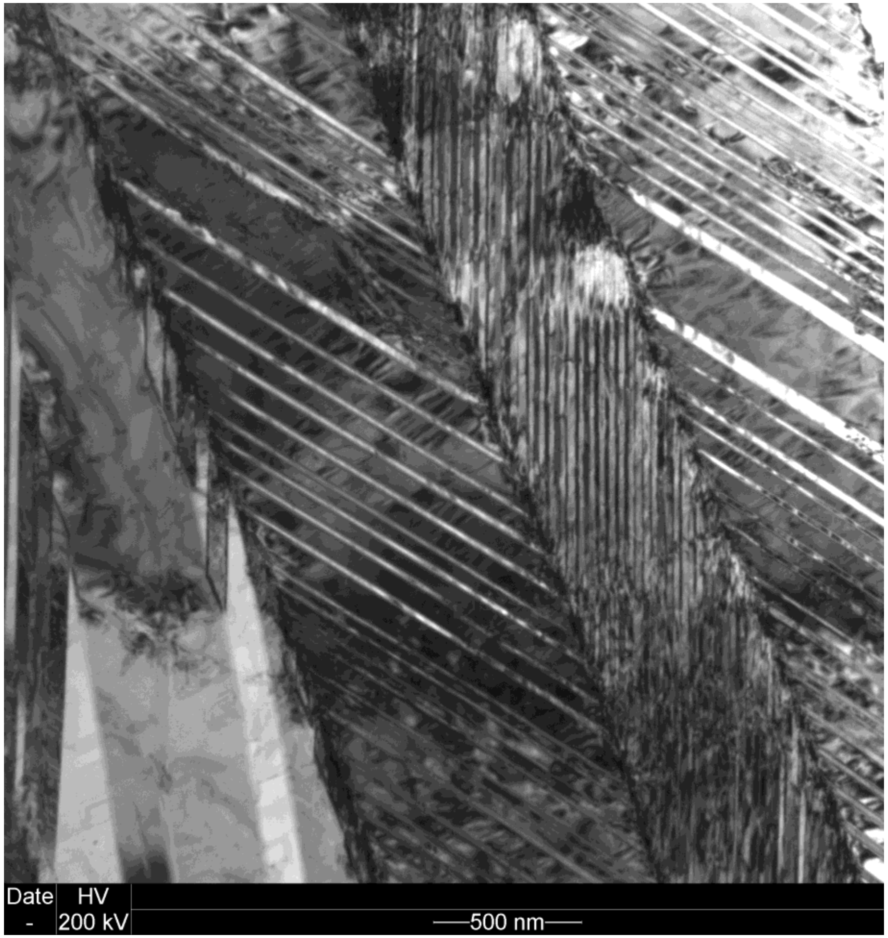
Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$)

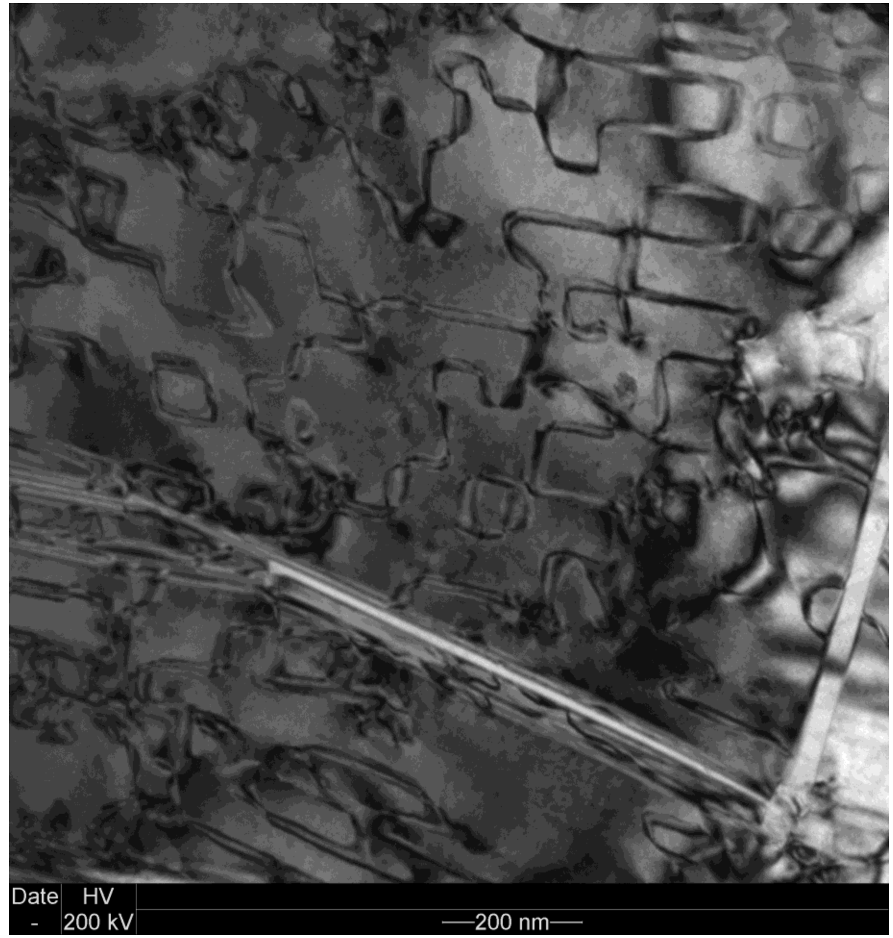


- ➢ **Extruded and aged**
- ➢ **No major aging effects (single phase)**

Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$)

TEM images show no precipitate phase (Ext. 159)



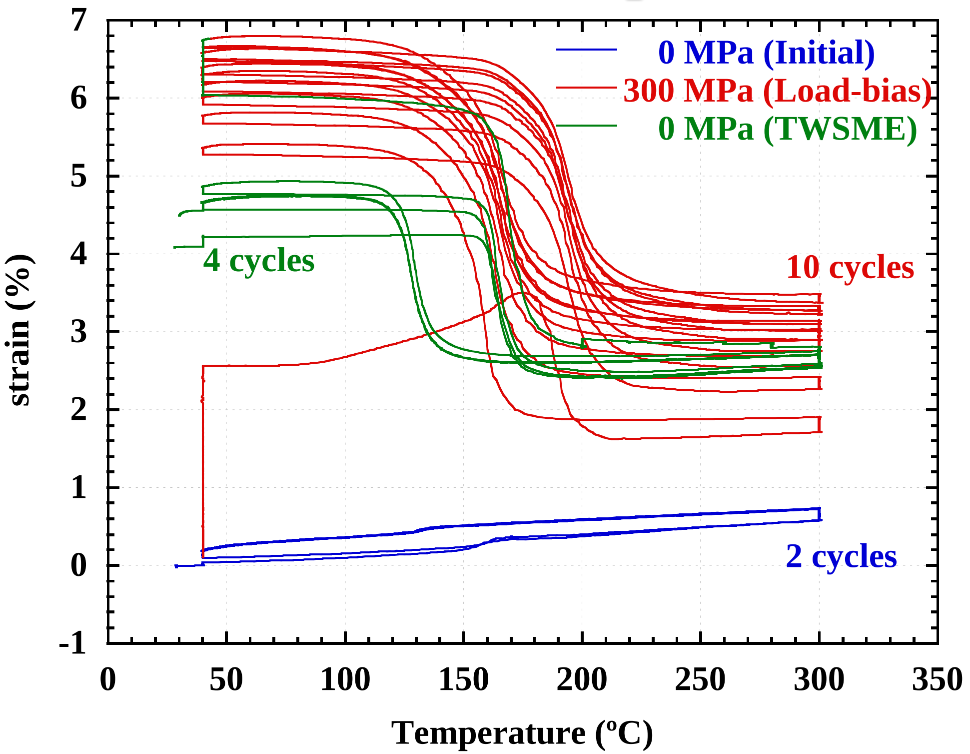


- Martensite phase

- Antiphase domain boundaries
- No precipitates

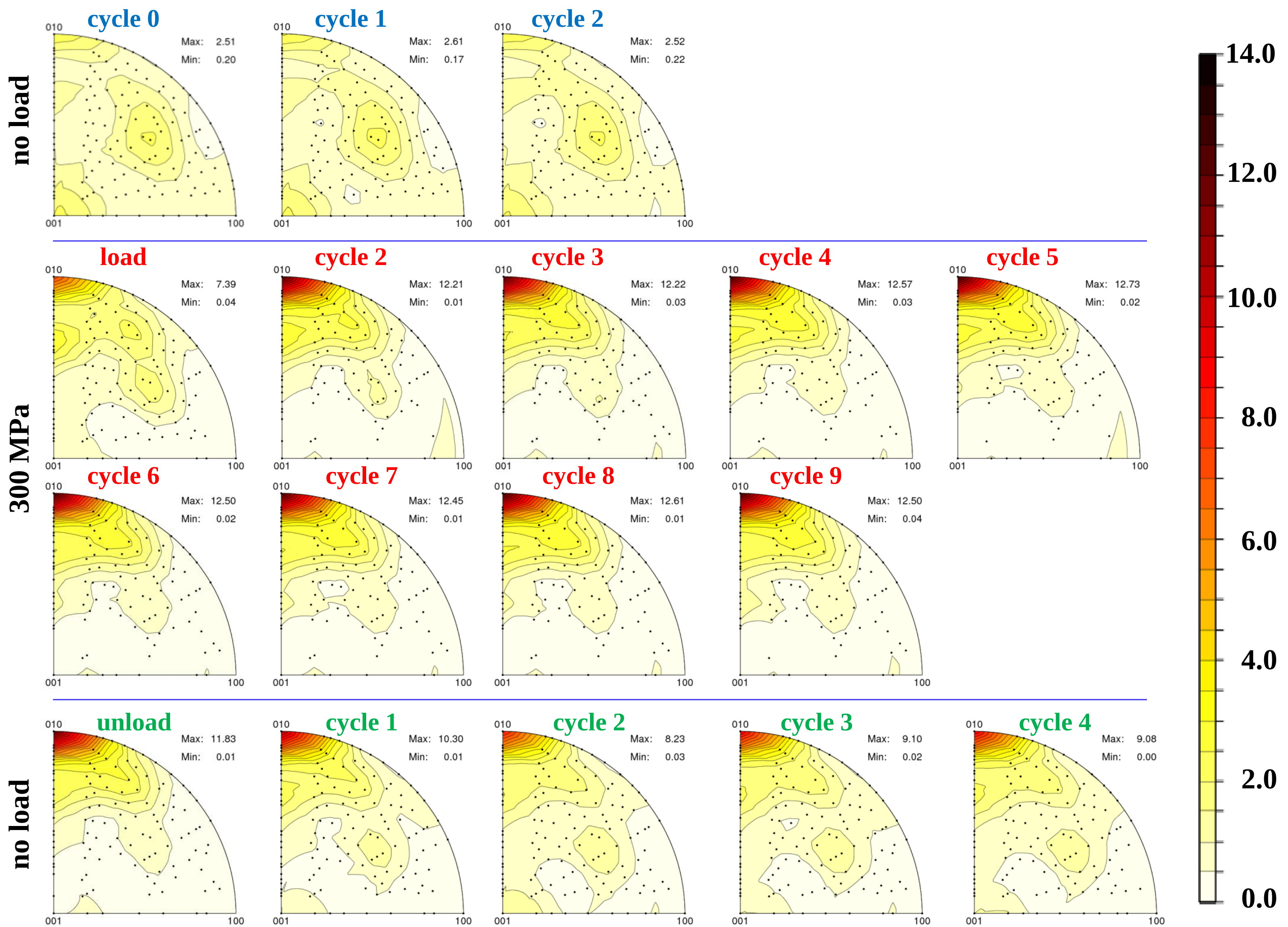
Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$) Isobaric Response

Macroscopic

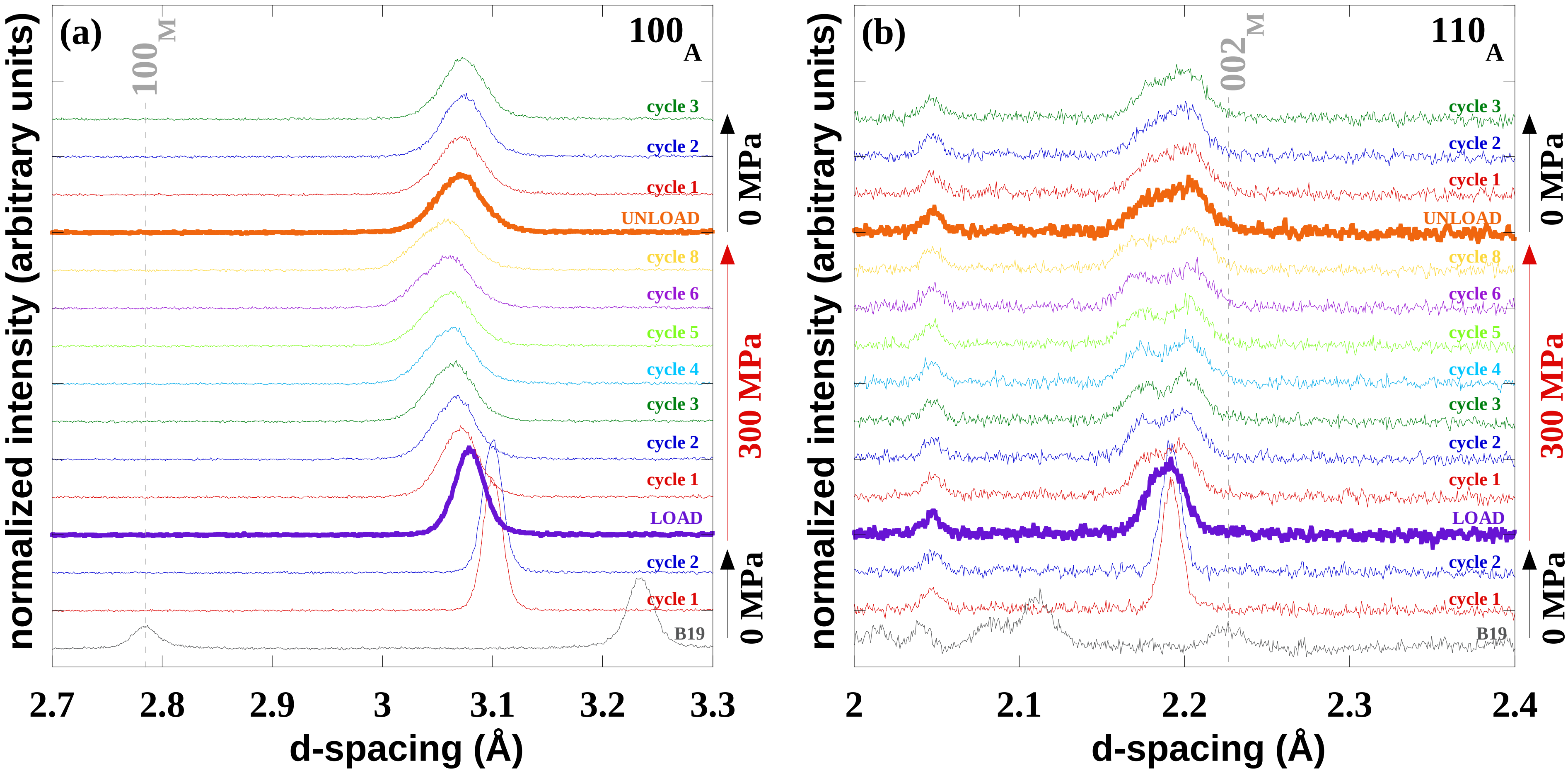


- 2 thermal cycles at 0 MPa
- 10 thermomechanical cycles at 300 MPa
- 4 thermal cycles at 0 MPa (TWSME)

Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$) TWSME
Texture Retained After Unloading



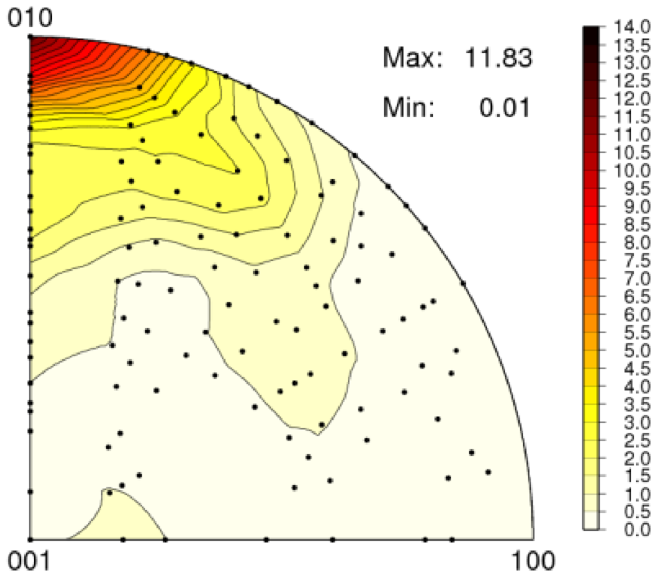
Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$) Isobaric Response
Retained Martensite at 300 °C



Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$) Summary HTSMA with TWSME

TEM

Neutron diffraction



- No Precipitates formed after aging at 400 °C
- Large amount of dislocations present after load-bias tests
- Stabilized twins at room temperature responsible for TWSME

HTSMAs Summary

- **Ni-Rich NiTiHf: Good stability**
  - **Neutron, X-ray and electron diffraction confirmed the formation of fine, nanometer size, coherent precipitates through careful stoichiometry control and aging. This precipitate phase is believed to be the stabilizing factor in this $Ni_{50.3}Ti_{29.7}Hf_{20}$ alloy**

- **Ni-Rich NiTiPd: Good TWSME**
  - **Composition control on the Ni(Pd)-Rich ($Ni_{24.3}Ti_{49.7}Pd_{26}$) resulted in a good TWSME, but unstable biased actuation**

- **Choice of alloy based on application:**
  - **Targeted alloy design to meet application requirement can be done to optimize properties**

- **Diffraction data served to provide a link between microscopic and macroscopic behavior, and supply information pertinent to the proper formulation of SMA micromechanics models**

Acknowledgment

- **NASA Fundamental Aeronautics Program, Aeronautical Sciences**
- **Basic Energy Sciences (DOE)**
- **Sandia National Laboratory**
- **Boeing & TAMU**

Thank you